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Description

This invention relates to a method of producing granular activated carbon using a chemical activation process.

Activated carbon, a porous adsorbent, is widely used in industry in the purification of liquids and gases. For example, a gas which is to be purified is passed through a bed of granular activated carbon. As the gas passes through the activated carbon bed, molecules of impurities in the gas are adsorbed onto the surface of the activated carbon. Consequently, the larger the surface area of the activated carbon the more efficient the filter will be in removing impurities.

Activated carbons of commercial importance can exhibit specific surface areas of up to $1500 m^2/g$ contained in a pore volume of approximately 1ml/g and to achieve this large surface area, pores of very small dimensions are involved. As pore size increases the surface area per unit volume decreases.

Pore sizes are defined as micropores, mesopores and macropores. Micropores and mesopores contribute to the adsorptive capacity of the granular activated carbon whereas the macropores merely reduce the density and are detrimental to the adsorbent effectiveness of the granular activated carbon.

Therefore, the pore structure of the carbon with its inherent surface area is of paramount importance in determining the effectiveness of the activated carbon as an adsorbent.

However, in the case of granular activated carbon the density is also an important feature of the effectiveness of the adsorbent, as the application of granular activated carbon is invariably in the form of a static bed of fixed volumetric size.

Chemically activated carbon, by virtue of its raw materials and manufacturing process, tends to be of low density with a highly developed mesopore structure. The latter feature is a desirable feature, the former a disadvantage of any granular form of chemically activated carbon. The success of any process to manufacture granular chemically activated carbon is dictated by its capacity to combine retention of the mesoporous nature with development of a high density by minimisation of macroporosity, which does not contribute to adsorptive effectiveness.

The normal method used to determine the efficiency of a granular activated carbon is the weight of material it can adsorb, per unit volume of activated carbon.

This test is normally carried out by placing a volume of activated carbon in a standard U-tube and passing a vapour through the activated carbon. The carbon is weighed before and after this process and the difference provides the weight of substance adsorbed by the carbon.

The raw material normally used in the production of chemically activated carbon is a carbonaceous vegetable material such as wood which has been milled to a 2-5mm particle size. The activated carbon when produced is usually either ground into a powder form for use in liquid purification or shaped into pellets of various sizes using a binder, for use in gas purification.

There are a number of such uses for activated carbons from the removal of coloured compounds present as impurities in the products of a chemical reaction to the purification of gases prior to discharge to the atmosphere. However, there are a number of problems inherent in the use of wood as a raw material to produce directly a chemically activated pelletised granular form.

The hollow fibrous structure of wood is such that it is impossible to produce a high density activated carbon from a wood flour raw material. The wood also lacks a natural binding agent, such as lignin, in sufficiently large quantities and an additional binding agent would have to be introduced in the production of the activated carbon to prevent the breakdown of the particle structure of the granular carbon during processing.

The cellular structure of wood is such that the granular activated carbon produced from it is capable of adsorbing a maximum of 6-7g of impurities/100ml of activated carbon due to its low density. This is below the figure required for a number of applications of activated carbon.

This is not of great importance in a powder liquid phase application as there is normally no strict limit on the volume of activated carbon which can be used.

However, as previously discussed in the case of granular application, there is an upper limit on the volume of activated carbon which can be used. Consequently, if the granular activated carbon is to be able to perform effectively then the volumetric adsorption factor (g/100ml) must be increased substantially by increase in the product density.

Therefore, the introduction of a method of producing a more efficient activated carbon would be extremely advantageous.

According to the present invention there is provided a method of producing activated carbon from a carbonaceous vegetable product starting material comprising the steps of:

a) comminuting said starting material to form particles;

b) mixing said comminuted particles of starting material with phosphoric acid to impregnate said particles with said acid;

c) pelletising said mixture of comminuted particles and phosphoric acid to form pellets; and

d) carbonising said treated particles,

characterised in that said comminuted particles have a mean particle size of greater than 30µm and less than 60µm and that said carbonaceous vegetable product starting material has a concentration of natural binding agent which is greater than 30% by weight.

Nut shell, fruit stone and kernel, and in particular, olive stone, almond shell and coconut shell are especially useful and contain high levels of natural binding agents, for example, lignin.

These materials, when ground to a particle size of 2-5 mm as normally used with the chemical activation process, are incapable of absorbing sufficient quantities of the activation chemical and produce inferior quality activated carbon. However, by careful milling and classification, a particle size distribution results which not only allows absorption of sufficient activation chemical but also maximises the density of the resulting carbonized product.

The starting material is comminuted to form particles of a mean particle size greater than 30µm and less than 60µm. The most preferable mean particle size being 40µm.

Preferably, the treated particles are mixed in a mechanical mixer.

Preferably, the said phosphoric acid is 60-80% ortho phosphoric acid and is added to the particles in a weight ratio of between 1.0:1 and 1.8:1 respectively and preferably a ratio of 1.4:1.

Preferably, the treated particles of starting material are pelletised by a rotary pelletiser.

Preferably, the pellets undergo heat treatment to remove water and other volatile constituents present in the pellets to consolidate the granular nature.

Preferably, the heat treatment comprises heating at a temperature of approximately 120°C for a duration of between 10 and 40 minutes.

Preferably, the heat treated pellets are carbonised thus producing activated carbon.

Most preferably, the said pellets are carbonised at a temperature between 400°C and 500°C for a duration of between 15 minutes and 20 minutes.

Preferably, the remaining activation chemical is washed from the pellets and recycled for repeated use.

Preferably, the pellets of activated carbon are dried.

Further according to the present invention there is provided activated carbon produced by the method of the invention.

An embodiment of the present invention will now be described, by way of example, with reference to the accompanying drawing which is a flow chart of the steps involved in a method of producing activated carbon in accordance with the present invention.

As a starting material there is selected a young carbonaceous vegetable product which is high in natural binding agents. The binding agent is lignin and starting materials having a concentration greater than 30% by weight are suitable. One such starting material is olive stones which have a concentration of over 36% lignin by weight. Other starting materials which have a lignin concentration of greater than 30% are almond shell at 30.7% and coconut shell at 34.3%. The embodiment hereafter described relate to the use of olive stones.

Olive stones are ground into particles of a mean particle size of between 30µm and 60µm; the most preferable mean particle size being 40µm. Phosphoric acid (67%) is added to the olive stone particles in a weight ratio of 1.4:1 respectively. Reducing the olive stones to particles of this size before adding the phosphoric acid is important as otherwise the phosphoric acid could not impregnate the olive stones, due to the relatively high density of the stones.

The treated material is mixed in a mechanical mixer for between 15 to 45 minutes.

The phosphoric acid impregnated particles are then pelletised by a rotary pelletiser into pellets of approximately 2mm to 5mm length and between 1.0mm to 3.0mm diameter.

The said pellets are then heated at approximately 120°C for a duration of between 10 and 40 minutes and preferably for 20 minutes. Heating of the pellets in this way minimises the entrapment of bubbles of escaping volatile material during subsequent carbonisation and consolidate the granular form.

The pellets then undergo carbonisation in which they are heated to a temperature between 400°C and 500°C for a duration of between 15 minutes and 20 minutes, thus producing activated carbon.

The activated carbon is then washed to recover any residual phosphoric acid and the said phosphoric acid is then recycled for re-use in the process.

The granular chemically activated carbon is then dried to remove water, and classified according to the size of the pellets.

The final product of pelletised chemically activated carbon is then packaged for distribution.

Modifications and improvements may be incorporated without departing from the scope of the invention.

Claims

1. A method of producing activated carbon from a carbonaceous vegetable product starting material comprising the steps of:

a) comminuting said starting material to form particles;
b) mixing said comminuted particles of starting material with phosphoric acid to impregnate said particles with said acid;
c) pelletising said mixture of comminuted particles and phosphoric acid to form pellets; and
d) carbonising said treated particles,
characterised in that said comminuted particles have a mean particle size of greater than 30µm and less than 60µm and that said carbonaceous vegetable product starting material has a concentration of natural binding agent which is greater than 30% by weight.

2. A method as claimed in Claim 1 wherein the starting material is a nutshell.

3. A method as claimed in Claim 1, wherein the starting material is a fruit stone.

4. A method is claimed in either Claim 1, wherein the starting material is a kernel.

5. A method as claimed in Claim 3, wherein the starting material is olive stone.

6. A method as claimed in Claim 2, wherein the starting material is almond shell.

7. A method is claimed in Claim 2, wherein the starting material is coconut shell.

8. A method as claimed in any one of Claims 1 to 7, wherein the natural binding agent is lignin.

9. A method as claimed in any one of Claims to 8, wherein the mean particle size is 40µm.

10. A method as claimed in any one of Claims 1 to 9, wherein the said phosphoric acid is 60-80% ortho phosphoric acid and is added to the particles in a weight ratio of between 1.0:1 and 1.8:1 respectively.

11. A method as claimed in Claim 10, wherein the ratio is 1.4:1.

12. A method as claimed in any one of Claims 1 to 11, further comprising a drying treatment to remove water and other volatile constituents present in the pellets to consolidate the granular nature before carbonisation.

13. A method as claimed in Claim 12, wherein the heat treatment comprises heating at a temperature of approximately 120°C for a duration of b tween 10 and 40 minutes.

14. A method as claimed in Claim 13, wherein the treated particles are carbonised at a temperature between 400°C and 500°C for a duration of between 15 minutes and 20 minutes.

Patentansprüche

1. Verfahren zum Herstellen von Aktivkohle aus einem kohlenstoffhaltigen Pflanzenprodukt als Ausgangsmaterial, bestehend aus den Stufen:
a) Verkleinerung des Ausgangsmaterials zur Ausbildung von Teilchen;
b) Mischen der verkleinerten Teilchen des Ausgangsmaterials mit Phosphorsäure zur Imprägnierung der Teilchen mit dieser Säure;
c) Pelletisieren des Gemisches der verkleinerten Teilchen und der Phosphorsäure zur Ausbildung von Pellets; und
d) Verkohlen dieser behandelten Teilchen,
dadurch gekennzeichnet, daß die verkleinerten Teilchen eine mittlere Teilchengröße von mehr als 30 µm und weniger als 60 µm haben, und daß das kohlenstoffhaltige Pflanzenprodukt als Ausgangsmaterial eine Konzentration an natürlichem Bindemittel von mehr als 30 Gew.-% hat.

2. Verfahren nach Anspruch 1, bei welchem das Ausgangsmaterial eine Nußschale ist.

3. Verfahren nach Anspruch 1, bei welchem das Ausgangsmaterial ein Obstkern ist.

4. Verfahren nach Anspruch 1, bei welchem das Ausgangsmaterial ein Kern ist.

5. Verfahren nach Anspruch 3, bei welchem das Ausgangsmaterial ein Olivenkern ist.

6. Verfahren nach Anspruch 2, bei welchem das Ausgangsmaterial eine Mandelschale ist.

7. Verfahren nach Anspruch 2, bei welchem das Ausgangsmaterial eine Kokosnußschale ist.

8. Verfahren nach einem der Ansprüche 1 bis 7, bei welchem das natürliche Bindemittel Lignin ist.

9. Verfahren nach einem der Ansprüche 1 bis 8, bei welchem die mittlere Teilchengröße 40 µm ist.

10. Verfahren nach einem der Ansprüche 1 bis 9, bei welchem die Phosphorsäure eine 60-80% Orthophosphorsäure ist und den Teilchen mit einem Gewichtsverhältnis zwischen 1.0:1 und 1.8:1 hinzugefügt ist.

11. Verfahren nach Anspruch 10, bei welchem das Verhältnis 1.4:1 ist.

12. Verfahren nach einem der Ansprüche 1 bis 11, welches weiterhin eine Trocknungsbehandlung zur Entfernung von Wasser und anderen flüchtigen Bestandteilen aufweist, die in den Pellets vorhanden sind, um die körnige Beschaffenheit vor dem Verkohlen zu festigen.

13. Verfahren nach Anspruch 12, bei welchem die Wärmebehandlung ein Erwärmen bei einer Temperatur von etwa 120°C für eine Dauer zwischen 10 und 40 Minuten einschließt.

14. Verfahren nach Anspruch 13, bei welchem die behandelten Teilchen bei einer Temperatur zwischen 400°C und 500°C für eine Dauer zwischen 15 Minuten und 20 Minuten verkohlt werden.

Revendications

1. Procédé de fabrication de charbon actif à partir d'un matériau de départ à base de produit végétal carboné comprenant les étapes suivantes :
   a) pulvériser ledit matériau de départ en vue de former des particules ;
   b) mélanger lesdites particules pulvérisées de matériau de départ à de l'acide phosphorique afin d'imprégner lesdites particules avec ledit acide ;
   c) agglomérer ledit mélange de particules pulvérisées et d'acide phosphorique afin d'obtenir des granulés ; et
   d) carboniser lesdites particules traitées,
   caractérisé en ce que la granulométrie moyenne desdites particules pulvérisées est comprise entre 30 µm et 60 µm et en ce que le matériau de départ à base de produit végétal carboné a une teneur en agent liant naturel supérieure à 30 % en poids.

2. Procédé selon la revendication 1, caractérisé en ce que le matériau de départ consiste en des coquilles de noix.

3. Procédé selon la revendication 1, caractérisé en ce que le matériau de départ consiste en des noyaux de fruit.

4. Procédé selon la revendication 1, caractérisé en ce que le matériau de départ consiste en des amandes de noyau ou de fruit.

5. Procédé selon la revendication 3, caractérisé en ce que le matériau de départ consiste en des noyaux d'olive.

6. Procédé selon la revendication 2, caractérisé en ce que le matériau de départ consiste en des coquilles d'amande.

7. Procédé selon la revendication 2, caractérisé en ce que le matériau de départ consiste en des noix de coco.

8. Procédé selon l'une quelconque des revendications 1 à 7, caractérisé en ce que l'agent liant naturel est de la lignine.

9. Procédé selon l'une quelconque des revendications 1 à 8, caractérisé en ce que la granulométrie moyenne des particules est de 40 µm.

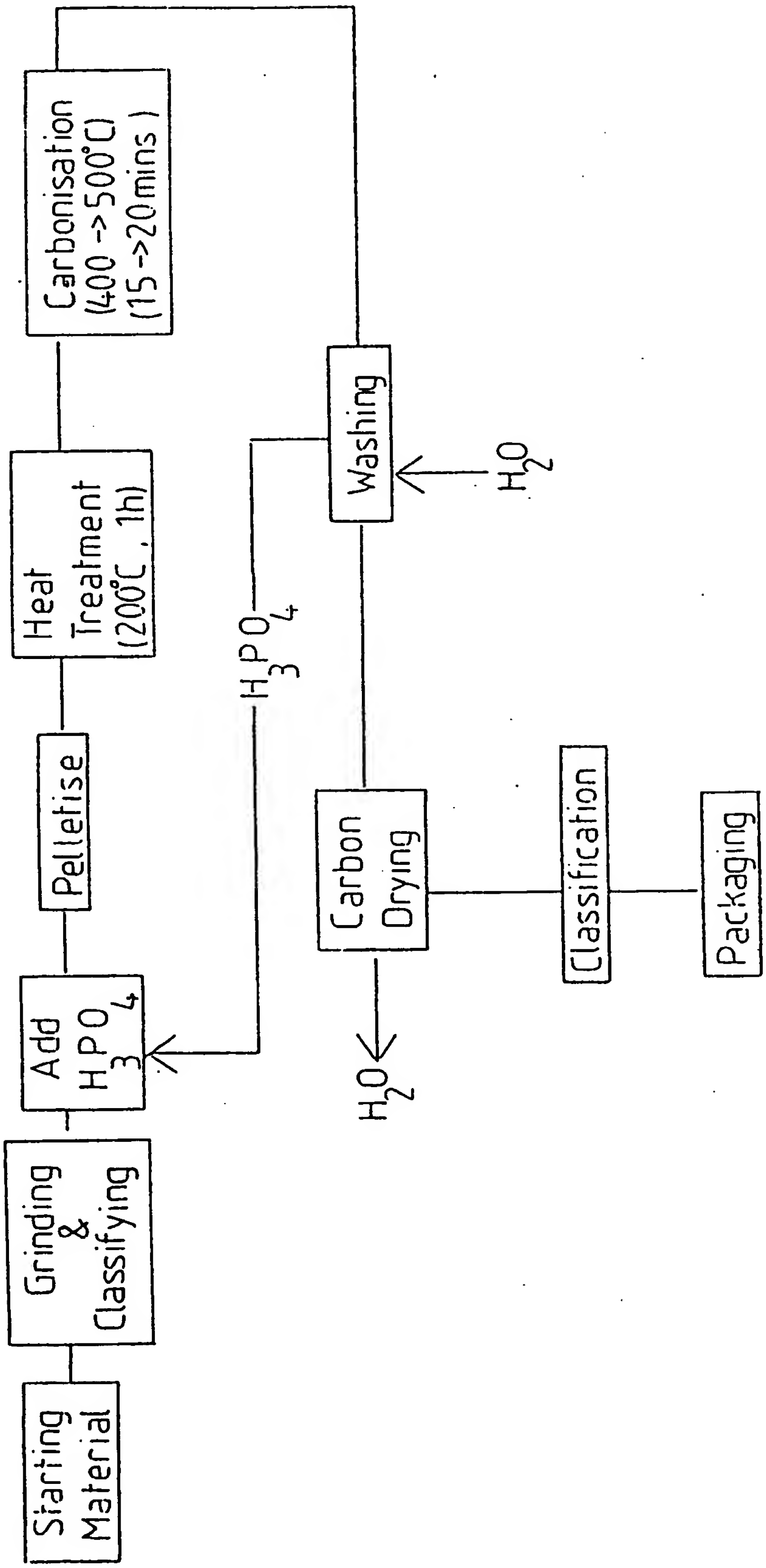
10. Procédé selon l'une quelconque des revendications 1 à 9, caractérisé en ce que ledit acide phosphorique est de l'acide ortho-phosphorique à 60-80 % et est respectivement ajouté aux particules selon un rapport pondéral compris entre 1,0:1 et 1,8:1.

11. Procédé selon la revendication 10, caractérisé en ce que le rapport de mélange est de 1,4:1.

12. Procédé selon l'une quelconque des revendications 1 à 11, caractérisé en ce qu'il comprend également un traitement de séchage visant à éliminer l'eau et les autres composants volatils présents dans les granulés afin de consolider la structure granulaire avant carbonisation.

13. Procédé selon la revendication 12, caractérisé en ce que le traitement thermique comprend le chauffage à une température d'environ 120°C pendant une durée comprise entre 10 et 40 minutes.

14. Procédé selon la revendication 13, caractérisé en ce que les particules traitées sont carbonisées à une température comprise entre 400°C et 500°C pendant une durée comprise entre 15 et 20 minutes.


Starting Material
Grinding & Classifying
Add H_3PO_4
Pelletise
Heat Treatment (200°C, 1h)
Carbonisation (400 -> 500°C) (15 -> 20 mins)
Washing
H_2O
H_3PO_4
Carbon Drying
H_2O
Classification
Packaging